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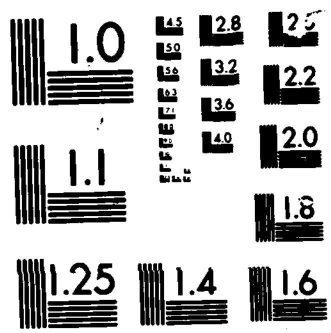

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TECHNICAL REPORT ARAED-TR-86011

SCANNING ELECTRON MICROSCOPE STUDY OF NITROGUANIDINE, M31A1, AND M30 PROPELLANTS

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US ARMY
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U. S. ARMY ARMAMENT RESEARCH AND DEVELOPMENT CENTER
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Scanning electron photomicrographs were made of a reference sample of nitroguanidine (NQ) obtained from a Canadian manufacturer who has suspended production. Similar photos of nitroguanidine made at the Sunflower Army Ammunition Plant (SFAAP) and at the U.S. Army Armament Research and Development Center (ARDC) were compared to the Canadian reference material.

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20. ABSTRACT (continued)

From these photomicrographs the range of crystal sizes in the different samples was estimated. These NQ samples were incorporated into M31A1 and M30 propellants. Scanning electron microscope (SEM) photomicrographs of the resultant propellants revealed the long NQ needles break up during processing and that particle size differences in the original NQ are also seen in the same order in finished products.

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INTRODUCTION

Canadian nitroquanidine (NQ) used in triple base propellant ceased to be manufactured in 1978. In order to provide a replacement for this critical material, manufacture was initiated at the Sunflower Army Ammunition Plant (SFAAP). A critical part of the process is control of particle size. A difficult problem in this regard is the on-stream measurement of the particle size. Nor is it a trivial matter to measure the particle size of the end product. Comprehensive studies have been made of the best means for making both types of particle size measurements. The Fisher Sub Sieve Sizer (FSSS) is the current specification method used to measure the dry product. Many other methods have been investigated, too. Although classical particle size measurements with the microscope are tedious as compared to other automated instrumental techniques, they still are the most accurate ones.

Hence, a scanning electron microscope (SEM) study was undertaken to get an accurate assessment of the particle size of both the old Canadian product as reference and different batches of product from the SFAAP as well as several experimental ones made at the U.S. Army Armament Research and Development Center (ARDC). This report is concerned with the findings of this study.

EXPERIMENTAL

Canadian, Sunflower Army Ammunition Plant (SFAAP), and experimental batches of NQ were examined. The latter types had been prepared in small lots of up to 200 pounds each at this installation. An AMRAY VTC 1400 SEM was used to photograph the samples; Dow Polystyrene Latex spheres with a diameter of 0.264 μm were used to calibrate the SEM's magnification scale. Polaron Instruments Microstick 1214 was used to mount samples on aluminum stubs for plating with Pd/Au prior to introduction into the SEM.

RESULTS AND DISCUSSION

Evaluation of NQ Samples

Measurements of the dimensions of crystals from SEM photomicrographs are recorded in table 1. The discontinued Canadian product, sample 1, CCL-9-418, is more uniform in particle size than the other samples. The two most uniform batches of American manufacture are sample 2, Sunflower SOW 83 H001-004, and sample 3, SOW 83 G001-002. Cursory inspection of photos at the same magnifications, figures 1 versus 2 and 3 versus 4 confirms that the crystals in the Canadian sample designated as 6 μm size are, on the whole, larger than those in the Sunflower sample designated as 4 μm size, SOW 83 H001-004. Thus SEM observations are in accord with previous relative (but not necessarily absolute) particle size designations arrived at by FSSS analysis. The white bar in the middle

of the legend at the bottom of the picture is a measurement reference labeled in μm . In the case of figures 1 and 2 the bar represents 100 μm ("U" in the legend). Although the Canadian sample contains smaller crystals which are about 10 μm in length and 0.25-0.5 μm wide, it is essentially free from "fines." The bulk of the needles are 30 to 150 μm in length and 5 to 15 μm wide. No distinction is made between width and thickness of the particles since, in most cases, only one side is seen in the photos. There may well be a tendency, at least in the case of the larger needles, for them to be more broad than thick.

The SOW 83H 001-004 NQ designated as 4 μm in size consists, for the most part, of fairly well-formed needles. However, some are fused together in bundles. Lengths of the needles vary from about 10 to 100 μm with a few being even longer. The bulk of them are nearer to being 100 μm long, with a relatively small amount of fines being present. The smaller needles tend to be about 0.25 μm in width and thickness whereas the larger ones are nearer 0.5 to 1.5 μm . Thickness and widths are nearly equal, though there may be a tendency for them to be somewhat more broad than thick. Appearance of the samples indicates that the crystallization process was well-controlled, giving a reasonably uniform product. Photomicrographs taken at higher magnifications are shown in figures 5 and 6.

The nominally 6 μm NQ, SOW 83G 001-002, contains noticeably larger sized needles than the 4 μm sample (figure 7 versus 8). The amount of fines in this 6 μm sample is moderate. The needles here are well-formed too, and sufficiently physically uniform to indicate the crystallization process was well-controlled. The fines consist of rods about 5 to 20 μm long and about 0.25 μm in width. The larger ones are up to about 300 μm in length and 2.5 to 10 μm in thickness and width. Again it appears that some of the larger crystals are wider than they are thick. There is a greater variation in the size of particles and incidence of larger ones in this sample than in the reference 6 μm Canadian one, CCL-9-418. By inference, the crystallization process was not as precisely controlled as the Canadian one.

The sample of NQ, SOW 83H 001-003, classed as 10 μm size contains significant numbers of needles that are larger than those in the preceding 6 μm sample (figure 9 versus 10). In this case, too, the appearance of the crystals indicates the process used to form them was relatively well controlled. However, the difference in size between the fines and large crystals is greater than in the case of the aforementioned 4 and 6 μm samples. The "fines" are rods ranging in length up to 50 μm. They are about 0.25-0.5 μm wide and probably of nearly equal thickness. The larger ones are as long as 1500 μm, in some cases, but more often 150 to 500 μm. They vary in width from about 10 to 20 μm. Though many appear to be of similar width and thickness, there are some that seem to be more broad than thick. Figure 11 shows the appearance of these nominally 10 μm crystals at a higher magnification.

The sample of NQ from ARDC, number 5, table 1, designated as LCP-A-6814, batch 1, is quite different in particle size and shape than the reference batch from Canada and the three from SFAAP. The latter four Canadian and Sunflower samples are nominally designated as being 4 to 10 μm in particle size. Considerably higher magnifications were required to characterize these ARDC samples with high concentrations of ultrafine particle size crystals. Mangifications 10 to 50

times greater than those used with the Canadian and Sunflower samples were required to bring out the details of the ultrafine particles (figures 12 and 13). Even the smallest crystals in ARDC Sample 5, table 1, LCP-A-6814) show good development. They tend to be more rectangular or hexagonal in shape as the case may be, and less needle-like than those in the Canadian and SFAAP samples. Their striking features are that they are wider and thicker with respect to length than the others. Also there is quite a disparity in sizes. Most of them are very small, but well developed, being in some cases less than 0.5 μm long and about 0.25 μm thick to about 10 μm long and 2 μm thick. The larger ones are up to 170 μm long and 20 μm thick. Good crystal development in both small and large crystals shows the process was well-controlled, in spite of the size difference.

The other sample made at ARDC, number 6, table 1, LCAP 6814, batch 2, was similar to sample 5, batch 1. It too was notable not only in superior crystal development but also in the difference between the sizes and morphology of the large and small crystals. The huge difference in sizes between the small and large crystals can be seen in figures 14 through 18. The small ones were as short as 0.5 μm in length and 0.25 μm wide. Most were at least 2 μm long and 0.5 μm wide. The large ones ranged from about 50 to 200 μm in length and were 10 to 20 μm wide. Width and thicknesses seemed to be similar.

Sample 7, table 1, which was a special ARDC laboratory prepared NQ, was somewhat different from all the other samples. However, it resembled the ARDC samples more closely than those from Canada and Sunflower. Like the ARDC samples it contained two types of crystals. The first were small, well-formed ones with widths varying from about 0.5 to 1.0 μm and lengths from 1 to 5 μm. The second type of crystal was the bulkier, larger kind which was about 3 to 5 μm wide and 5 to 10 μm long. In addition there was a third kind of crystal, which was an elongated form of the smaller types of crystals. These were about 0.3 to 1 μm wide and 10 to 40 μm long. The crystals were well-formed. Figures 19 through 23 illustrate these details.

Evaluation of Various NQ's in M31A1 and M30 Propellants

It is understandably more difficult to obtain measurements of NQ crystal dimensions in a propellant than with the original NQ samples. A notable feature of the five lots of M31A1 propellants is the more uniform length of the larger NQ crystals in the propellant matrix than is the case with the neat NQ samples. This can be attributed to breakup of the longer needles to shorter lengths during the propellant mixing operation. Crystallinity of the samples of NQ remains good in the finished propellants. As can be seen from table 2, significant size differences seem to exist between the different samples with regard to NQ crystal size. It should be emphasized, however, that the extreme measurements of crystal sizes are made upon the relatively few crystals that can be observed more or less entirely in the photomicrographs. Although such measurements can be made accurately, in the absence of many more photos at suitable magnifications there is a substantial degree of uncertainty in assigning given values as being representative of the whole batch of propellant.

The appearance of the four M31A1 propellants made from SFAAP NQ with nominal particle sizes of 4 to 10 µm, 1 and 3-5, table 2, are seen in figures 24 through 27. The original magnification in this series of photographs before they were altered in size for publication was 500X. The bar in the middle of the legend of the pictures at the bottom, however, can be used as a reference for measurements in every case regardless of changes in picture size. Figures 28 through 31 show the same propellants at twice the mangification of the previous four photographs. In each of these series the nominal 4 µm size material in RAD 84B00E 148, sample 1, table 2, is smaller than that in the other three Sunflower propellants. It is difficult to find much difference in the overall appearance of the crystals in the other three samples 3, 4 and 5, table 2. Figures 32 through 35 represent the propellants made with nominally 6 µm Canadian NQ. A comparison of the propellants made from the SFAAP and from Canadian NQ indicates that crystals in the latter material are of exceptionally uniform, small particle size. Of all of the photographs presented here, figure 35 most clearly shows hollow NQ crystals. Hollow ones are marked with arrows. Some of the other crystals in the photograph that are not marked appear to be hollow, too.

Observations on the six M30 propellants are listed in table 3. Significant differences in NQ particle size and orientation in M30 propellants were detected. It was difficult, for the most part, to find crystals that were fully revealed in the propellant mass. The range of particle sizes listed in table 3 were made on the few that were accessible to measurement. The M30 propellants are shown in figures 36 through 41, which are at the same magnification and in the same serial order as in table 3. Although the fine particles in figure 41 of the propellant with ARDC precipitated NQ are difficult to distinguish at 200X, the larger particles show up clearly. The similarity in appearance of the M30 propellants made from nominal 6 µm Canadian and 4 µm Sunflower NQ is illustrated in figures 42 and 43.

CONCLUSIONS

Comparing the Canadian NQ to that made at SFAAP and ARDC, one finds that the Canadian material is unique in that it is exceptionally uniform in particle size and is relatively free of "fines." The particle size rankings of the NQ reported by SFAAP were confirmed by SEM.

It is obvious that the nominal particle sizes determined by other particle size measuring techniques do not reflect the actual sizes seen in the SEM photomicrographs.

A comparison of the three SFAAP NQ samples indicates that the 4 µm NQ is more uniform in crystal size than both the 6 µm and 10 µ sizes.

The ARDC samples, morphologically speaking, are substantially different from the Canadian and SFAAP NQ. There is a preponderance of ultrafine particle size material in the ARDC samples. The special laboratory prepared ARDC NQ has significant numbers of short thick crystals, very small in size, and the smallest particle size of all the NQ samples evaluated.

The results for the M31A1 and M30 formulations indicate that the NQ crystals in the propellants containing Canadian 6 μm and the Sunflower 4 μm NQ, respectively, appear to be smaller than the other NQ's. The most atypical propellant was the ARDC special sample wherein the finer particles were relatively indistinguishable at 200X, compared to the other samples. The SEM photographs of the M30 and M31A1 propellants revealed that the varied, long needled NQ's break-up during processing and are more uniform in length in the finished propellants.

RECOMMENDATIONS

It should be pointed out that wet samples suspended in liquids can be examined by appropriate types of electron microscopes or ones with special accessories. Thus, electron microscopes can be used to back-up on-stream measurements to get a quick assessment of the product without resorting to rigorous particle size measurements and counting procedures. The utility of the scanning electron microscope for sizing the final, dry product has been demonstrated and should be used as a reference standard to ensure the accuracy of other methods which are based on less direct measurements.

Table 1. Crystalline nitroguanidine

Sample	1 Source	2 Designation	3[a] Nominal particle size μm, (uniformity of particle size)	4[b] Smaller particles width × length, μm	5 Ratio of length to width of larger ones in column 4	6[b] Larger particles, width × length, μm	7 Ratio of length to width of larger ones in column 6
1	Canada	CCL-9-418	6 (good)	0.25–0.5 × 10	20:1	5-15 × 30-150	10:1
2	US SFAAP	SOW 83 H001-004	4 (fair)	0.25 × 10	40:1	0.5-1.5 × 1000	67:1
3	US SFAAP	SOW 83 G001-002	6 (fair)	0.25 × 5-20	4:1	2.5-10 × 300	30:1
4	US SFAAP	SOW 83 H001-003	10 (poor)	0.25 × 0.5-50	100:1	10-20 × 1500	75:1
5	ARDC	LCP-A-6814 Batch 1	- (poor)	0.25-2 × 0.5 - 10	5:1	20 × 170	8.5:1
6	ARDC	LCP-A-6814 Batch 2	- (poor)	0.25-1 × 0.5 - 6	6:1	10-20 × 50-200	10:1
7	ARDC	Special	- (poor)	0.5-1.0 × 1-5; 0.3-1.0 × 10-40	5:1 40:1	3-5 × 5 - 10	2:1

[a]This is the size reported from standard analysis by FSSS.

[b]These measurements were made from SEM photomicrographs.

Table 2. M31A1 propellant

Number	Source	Designation	Nominal size Nitroguanidine µm	Smaller particles, width × length, µm	Larger particles width × length, µm	Remarks
1	Sunflower	RAD 84B 000E 148	4	0.5-1 × 2-5	3-10 × 10-60	Preponderance of larger, parallel oriented xtals
2	Canadian CCL	RAD 80M 070077	6	2 × 5-10	1.5-10 × 10-40	Crystals seem to be more randomly oriented than in 3 and 4
3	Sunflower	RAD 84B 000E 146	6	2 × 2-20	10-15 × 60 - 100	Preponderance of larger, parallel oriented crystals
4	Sunflower (Macaroni pressed)	RAD 84B 000E 147	6	1 X 5-15	2-15 × 20-100	Preponderance of larger crystals, some random orientation
5	Sunflower	RAD 84B 000E 151	10	0.5-1 X 2-5	4-10 × 30-70	Some random orienting of crystals as in 2, preponderance of larger crystals

Table 3. M30 propellants

Number	Source	Designation	Nominal size nitro-guanidine, μm	Estimated range of particle sizes, width × length, μm	Remarks
1	CCL	RAD78K069903	6	10-90	Crystals fairly uniform in size, nearly parallel
2	SFAAP	RADC000E137	6	(5-16) × (58-126)	Crystals fairly uniform in size, nearly parallel
3	SFAAP	RADC000E138	6	(8-21) × (63-100)	Crystals fairly uniform with some oversized ones, nearly parallel
4	SFAAP	RADC00E139	4	(2-12) × (up to 20)	High magnification photo at 2000 X shows wide variance in crystal size, nearly parallel
5	SFAAP	RAD84C000E143	10	(8-21) × (37-2632)	Wide variation in particle size, nearly parallel
6	ARDC	RADC000E145	ppt.	(0.5-38) × (1.5-232)	Wide variation in particle size, random orientation

Figure 1. Canadian 6 μm CCL-9-418 nitroguanidine

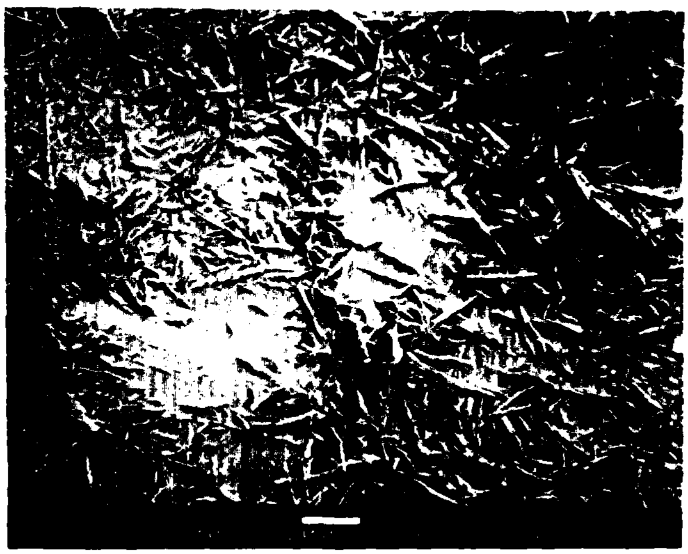

Figure 2. Sunflower 4 μm SOW 83 H001-004

Figure 3. Canadian 6 μm CCL-9-418

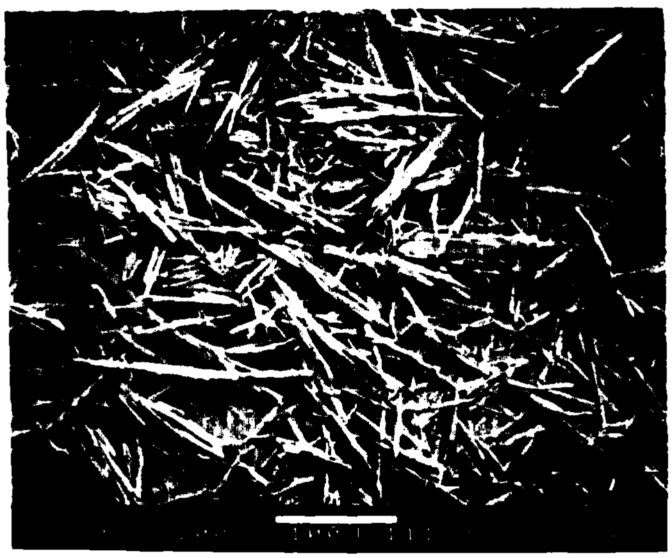

Figure 4. Sunflower 4 μm SOW 83H 001-004

Figure 5. Sunflower 4 μm SOW 83H 001-004

Figure 6. Sunflower 4 μm SOW 83H 001-004 at higher magnification than in figure 5

Figure 7. Sunflower 6 μm SOW 83G 001-002

Figure 8. Sunflower 4 μm SOW 83H 001-004

Figure 9. Sunflower 10 μm SOW 83H 001-003

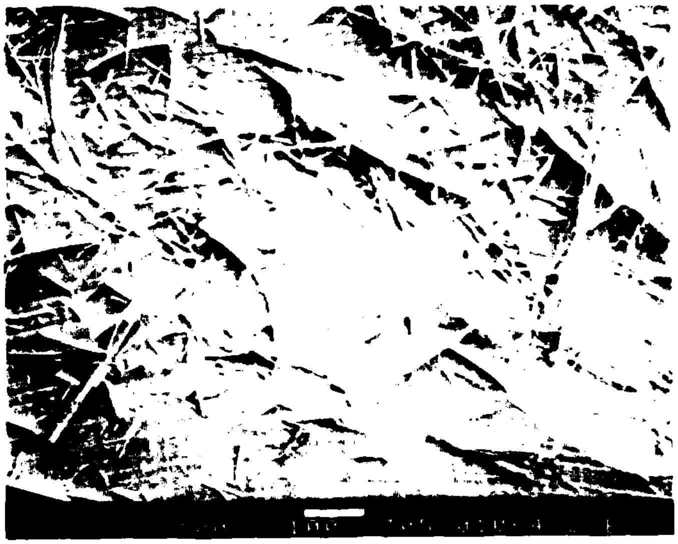

Figure 10. Sunflower 6 μm SOW 83G 001-002

Figure 11. Sunflower 10 μm SOW 83H 001-003

Figure 12. ARDC LCP-A-6814 Batch 1

Figure 13. ARDC LCP-A-6814 Batch 1 at higher magnification than in figure 12

Figure 14. ARDC LCP-A-6814 Batch 2 at even higher magnification than figures 12 and 13

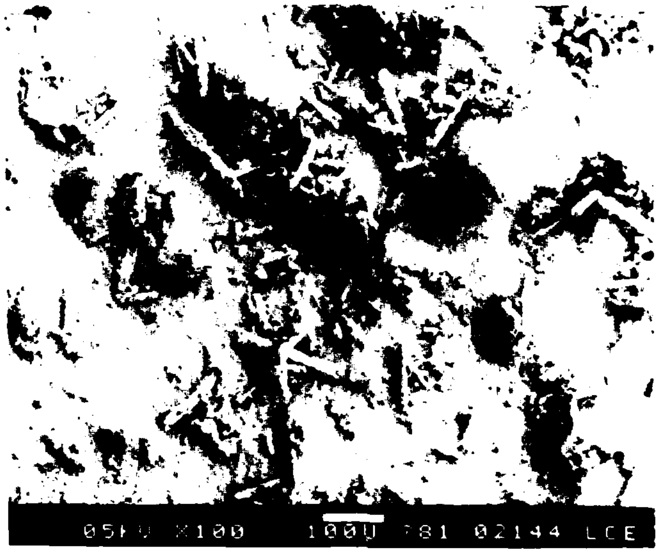

Figure 15. ARDC LCP-A-6814 Batch 2

Figure 16. ARDC LCP-A-6814 Batch 2 at higher magnification than in figure 15

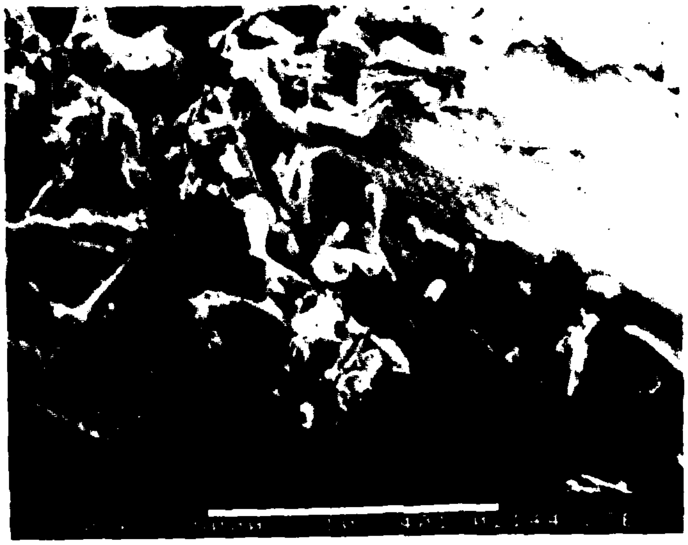

Figure 17. ARDC LCP-A-6814 Batch 2 at even higher magnification than in figure 16

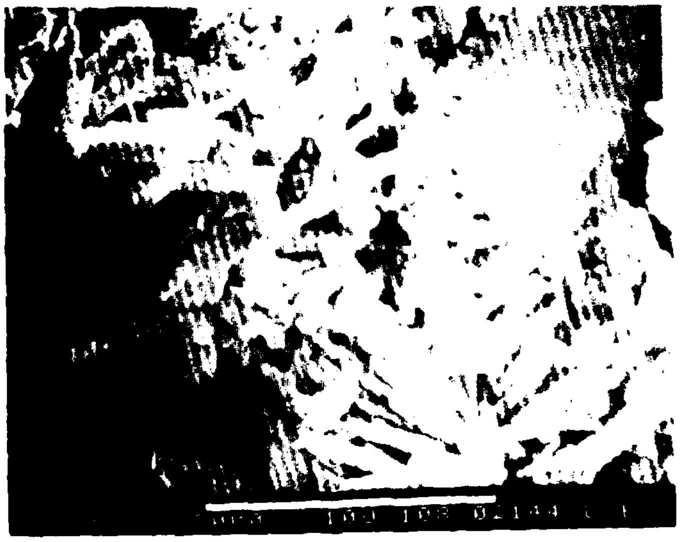

Figure 18. ARDC LCP-A-6814 Batch 2 at same magnification as in figure 17

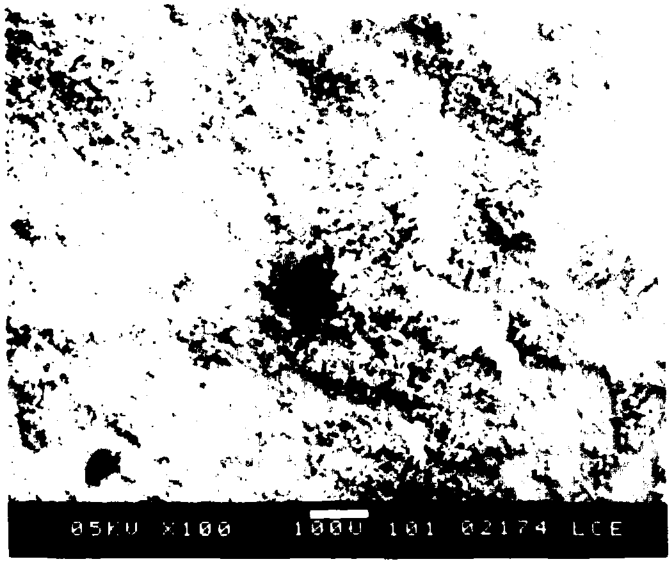

Figure 19. Special nitroguanidine

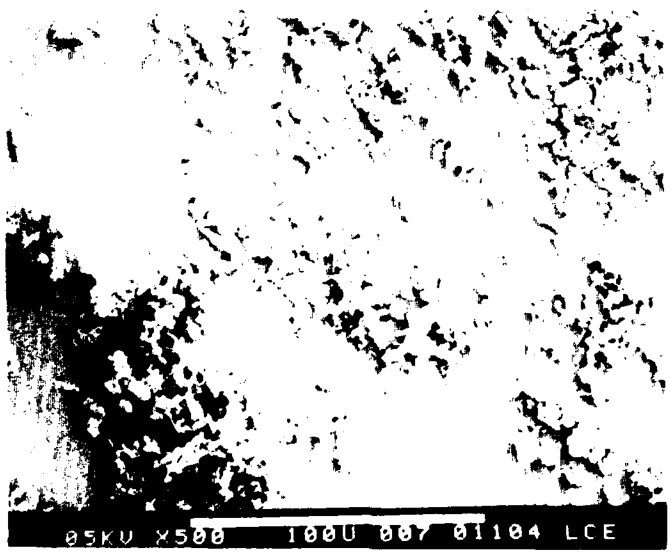

Figure 20. Special nitroguanidine at increased magnification than in figure 19

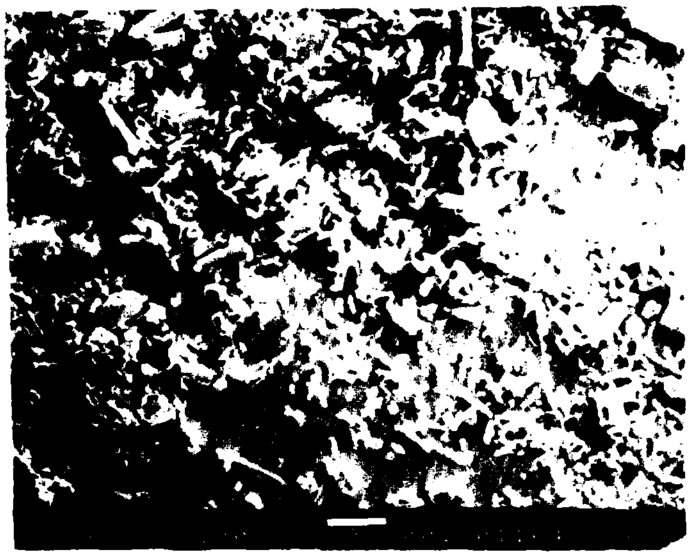

Figure 21. Special nitroguanidine at even higher magnification than in figure 20

Figure 22. Special nitroguanidine at even higher magnification than in figure 21

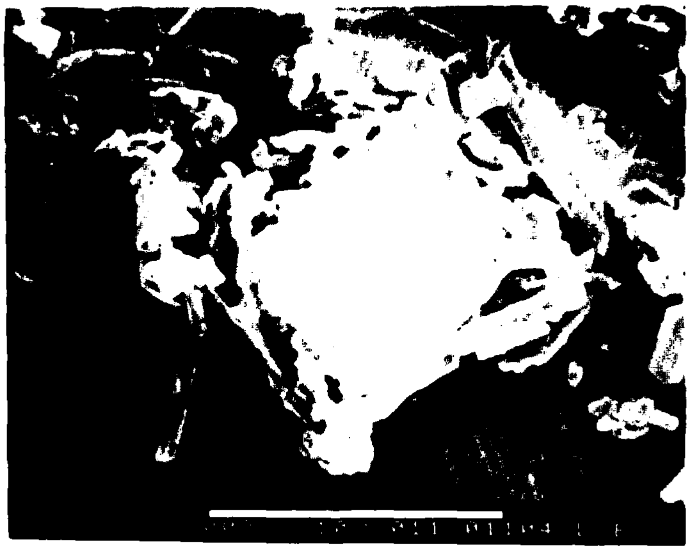

Figure 23. Special nitroguanidine at the highest magnification in the series of figures 19 through 23

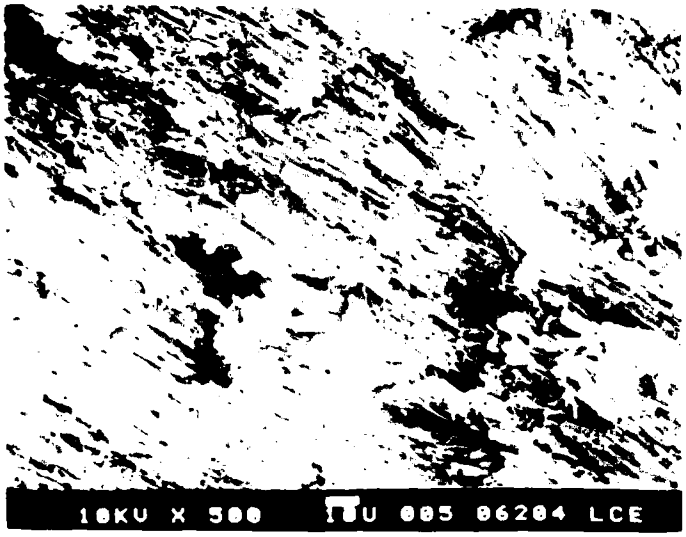

Figure 24. M31A1 4 μm RAD 84 B 000 E 148

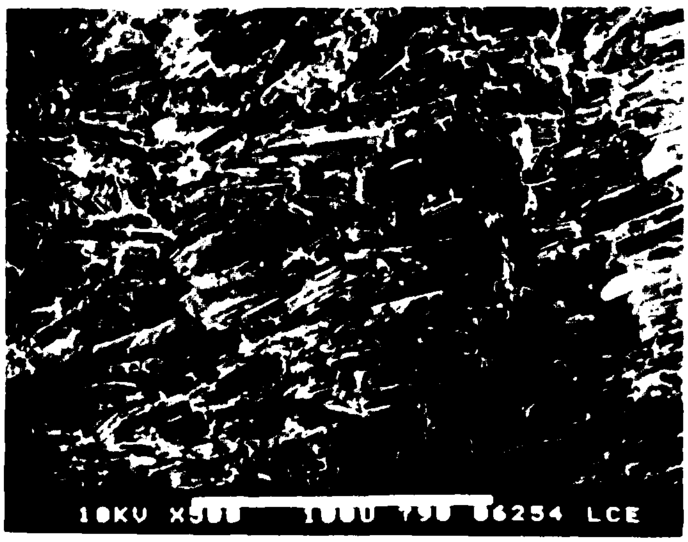

Figure 25. M31A1 6 μm RAD 84 B 000 E 146 in a different view from figure 24

Figure 26. M31A1 6 μm RAD 84 B 000 E 147 in another different view than those in figures 24 through 26

Figure 27. M31A1 10 μm RAD 84 B 000 E 151 in a view different from that in figure 32

Figure 28. M31A1 4 μm RAD 84 B 000 E 148

Figure 29. M31A1 6 μm RAD 84 B 000 E 146

Figure 30. M31A1 6 μm RAD 84 B 000 E 147

Figure 31. M31A1 10 μm RAD 84 B 000 E 151

Figure 32. M31A1 CCL 6 μm RAD 80 M 07 00 77

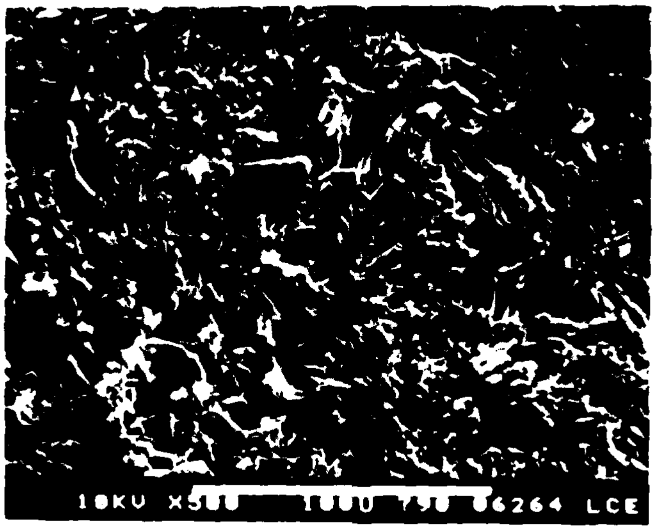

Figure 33. M31A1 CCL 6 μm RAD 80 M 07 00 77 in a view different from that in figure 32

Figure 34. M31A1 CCL 6 μm RAD 80 M 07 00 77 showing the fractured surface

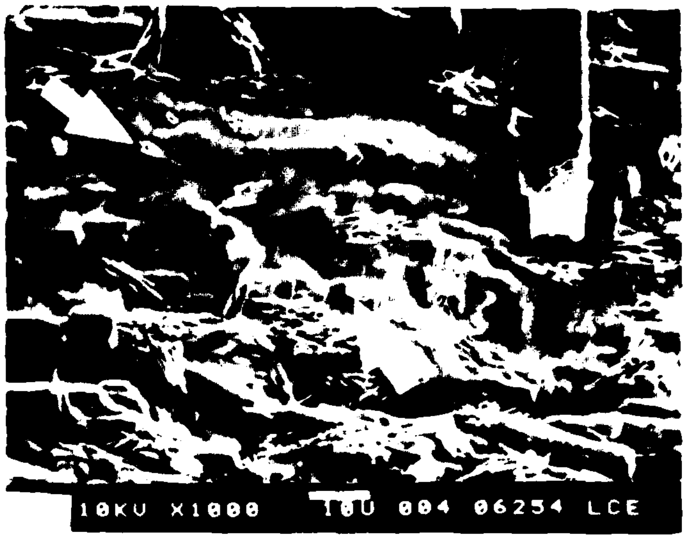

Figure 35. M31A1 CCL 6 μm RAD 80 M 07 00 77 showing the surface of the burning rate hole longitudinally from left to right

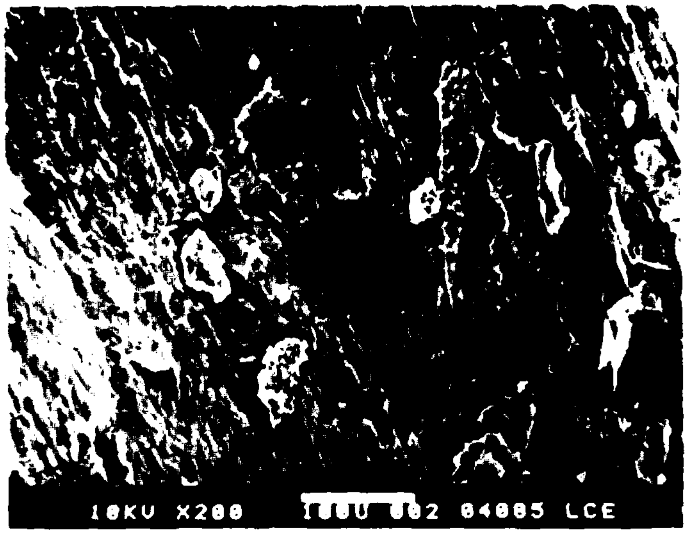

Figure 36. M30 CCL 6 μm RAD 78 K 06 99 03

Figure 37. M30 6 μm RAD C 000 E 137

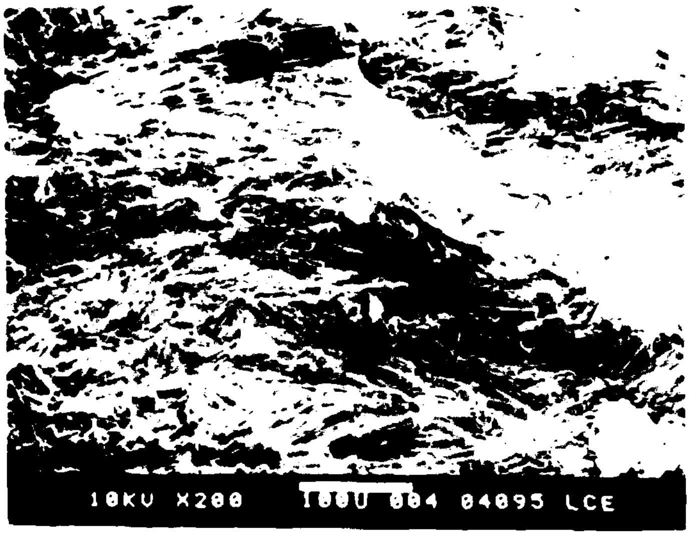

Figure 38. M30 6 μm RAD C 000 E 138

Figure 39. M30 4 μm RAD 84 C 000 E 139

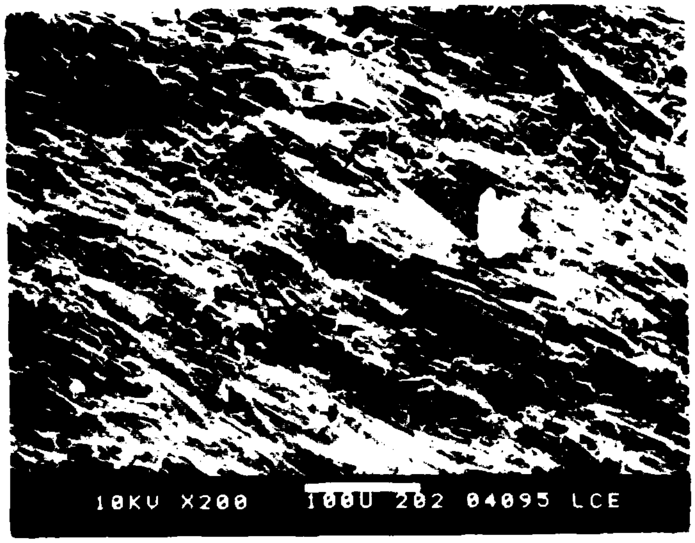

Figure 40. M30 10 μm RAD 84 C 000 E 143

Figure 41. M30 ppt. RAD C 000 E 145

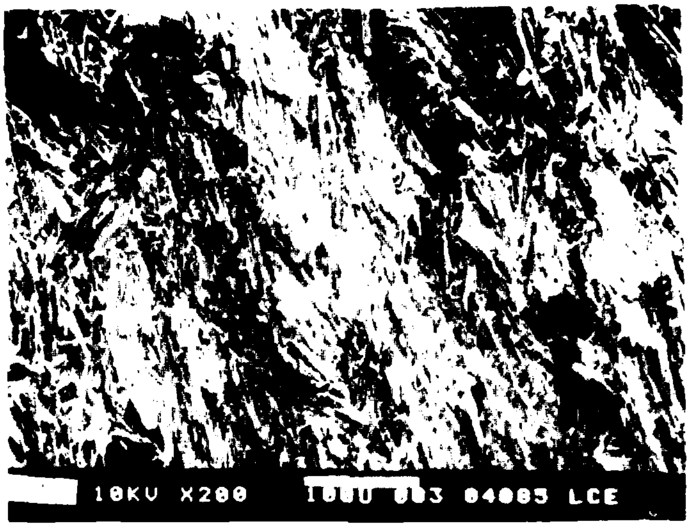

Figure 42. M30 CCL 6 μm RAD 78K 06 99 03

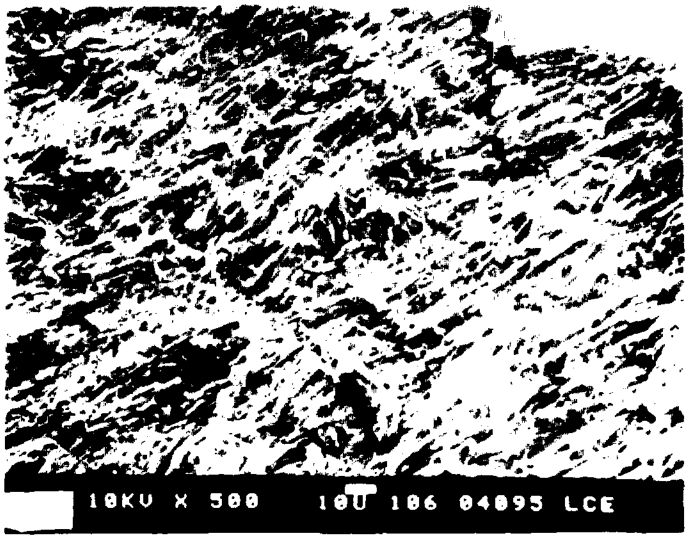

Figure 43. M30 4 μm RAD C 000 E 139

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